N64 24051 Code/ Cat. 3/ CR56457

Contract NAS 1-3244

RIGIDIZED INFLATABLE SOLAR ENERGY CONCENTRATOR

Third Quarterly Report

February - May 1964

# HUGHES

OTS PRICE

**XEROX** 

s \_ 2.60ph

MICROFILM

# HUGHES AIRCRAFT COMPANY AEROSPACE GROUP MATERIALS TECHNOLOGY DEPARTMENT Culver City, California

# RIGIDIZED INFLATABLE SOLAR ENERGY CONCENTRATIONS

Period of February 1964 to May 1964

by

S. Schwartz

## THIRD QUARTERLY PROGRESS REPORT

NASA Contract NAS 1-3244 NASA Control No. L-2923

Date of Publication May 1964

National Aeronautics and Space Administration Langley Research Center, Langley Station, Hampton, Virginia

APPROVED:

W.H. Colner, Manager

Materials Technology Department

Hughes Aircraft Company · Culver City, California

# CONTENTS

Abstra	act	•	•	•	•	•	•	•	•	•	•	•	•	1
Introd	uction			•		•	•	•	•	•	•	•	•	3
Polyes	ster Re	sin	Inves	tiga	ation		•	•	•	•	•	•	•	5
	Syntact	ic I	Foam		•	•	•	•	•	•	•	•	•	5
	Polyes	ter	Resin	ıs	•	•	•	•		•	•	•	•	7
	Shrinka	age	Tests	;	•	•	•	•	•	•	•	•	•	8
	Adhesi	on [	Γests		•	•	•	•	•	•	•	•	•	9
	Polyes	ter	Paral	bola	ıs	•	•	•	•	•	•	•	•	10
Ероху	Resin	Inv	estiga	tio	n	•	•	•	•	•	•	•	•	15
	Syntact	ic I	Foam		•	•		•	•	•	•	•	•	15
	Adhesi	on :	Tests		•	•		•		•	•	•	•	<b>i</b> 9
	''Specia	al''	Ероху	Re	esin	Tests		•	•	•	•	•	•	23
Mylar	Film	Γes	ts	•	•	•	•	•	•	•	•	•	•	25
Future	e Plans	!												27

# ILLUSTRATIONS

Figure 1.	Mylar surface showing a fabric pattern	1 1
Figure 2.	Patternless distortion surface, due to uneven shrinkage	12
Figure 3.	One-foot diameter Wyandotte polyester parabola with low resin content, fine weave cloth	1 4
Figure 4.	Typical blistered parabola	18

# TABLES

Table I.	Polyester shrinkage tests		•	9
Table II.	Polyester-primer adhesion tests	. •	•	10
Table III.	Blister and adhesion tests on epoxy synta foam		•	19
Table IV.	Cleaning techniques tests	•	•	21

#### **ABSTRACT**

24051

The investigation of polyester syntactic foams did not result in a satisfactory system. Solid, low shrink polyester resins were investigated, and two materials, Wyandotte PR 242 and FMC Dapon M, showed promise. Adhesion tests indicated that the polysulfide material, CS2414, is the best primer for polyester resins. The use of the lightest weight fabric, combined with as low a resin content as possible, results in the best optics with the polyester system.

Techniques were developed for distributing the epoxy syntactic foam evenly using a wedge type spreader bar. A series of tests was made to determine the causes and possible remedies for the Mylar blistering after post-cure. Evacuation before cure and a vacuum post cure give the best results. Two new epoxy hardener systems curing at 200 °F were investigated for use in the syntactic foam.

## INTRODUCTION

The principal objective of this program is to develop a technique for fabrication of a solar energy collector for use in a space environment. This collector should be fabricated as a compact package on Earth and should be capable of automatic inflation and rigidization in a space environment. The collector is to be made in a lenticular shape, one surface utilizing an aluminized Mylar film as the reflector, and the other surface a clear plastic to act simply as a pressurization member. After inflation to the desired shape the reflective surface would be rigidized by a pre-applied coating.

Preliminary tests of an ultraviolet radiation catalyzed polyester fiberglass rigidizing system indicated that this concept appeared feasible. The major problems in fabricating the paraboloid appear to be:
(1) production of a satisfactory bond from the rigid laminate to the Mylar film, (2) obtaining a satisfactory optical surface on the aluminized Mylar and (3) obtaining a satisfactory figure in the formed and rigidized Mylar.

In the work done during the first three months of the project it appeared that techniques could readily be devised to obtain a satisfactory bond to the Mylar. The optical surface produced by these techniques, however, was far from satisfactory in the preliminary tests. The major portion of the work done during the second three months therefore was devoted to techniques of improving the optical surface. It was found that the use of an epoxy syntactic foam gave an excellent optical surface, as well as satisfactory rigidization. During the ensuing quarter then, efforts were devoted to improvement of both the polyester rigidization system and the epoxy syntactic foam system.

#### POLYESTER RESIN INVESTIGATION

#### SYNTACTIC FOAM

Previous tests had established that rigidization by use of a syntactic foam results in a superior optical surface after cure. The first syntactic foams investigated utilized room temperature curing epoxy resin combined with phenolic microballoons. The resulting parabolas were rigid and lightweight and had excellent optical surfaces. However, attempts to use phenolic microballoons in conjunction with UV catalyzed polyester resins were unsuccessful because of the opacity of the phenolic fillers which prevented more than a surface cure. Substitution of glass microballoons for the phenolics was somewhat more successful in that initially a cure was obtained approximately .030 inch deep in a few hours. Because of these initial successes further efforts were made to develop a UV activated polyester syntactic foam which would cure throughout its thickness.

The first tests made with glass microspheres and polyester resins utilized silica "Siloons" distributed by the American Reinforced Plastics Company. This material was used in conjunction with a styrene based resin, American Cyanamid Laminac 4123, and benzoin as a catalyst. It was found with this combination that a rapid surface cure took place, followed by a further cure to a depth of approximately .030 inch. It was felt that the opacity of the particular microspheres might be causing the poor cure. Samples of Emerson and Cuming Eccospheres appeared to be more transparent when examined under a low power microscope. However, tests with the Laminac 4123 resin and the Eccospheres gave approximately the same results.

Tests were then made with a diallyl phthalate based resin, Hooker Chemical Company, Hetron 103, which, perhaps because of a slower curing rate, showed less tendency to a "skinning" effect and a more complete cure. The addition of benzoyl peroxide as a secondary catalyst, then resulted in a mixture which would cure nearly completely (.093 inch thick) in a 24-hour period left outdoors in the sunlight. The optics

obtained, while not quite as good as the epoxy samples, were nevertheless fair. A good figure was obtained (visually judged); however, a good deal of local "patternless" distortion was found. The formulation which was found to be the most satisfactory, was as follows:

Hetron 103 resin	100 pts
Eccospheres	30 pts
Benzoin - TCP mixture	2 pts
Benzoyl peroxide — TCP	2 pts
Cab-O-Sil	3 pts

This mixture (excluding the glass beads) was found to show very little signs of gelation, for as long as two weeks. With the addition of the glass microspheres the mixture showed signs of gelation in 24 hours. However, the mixture did remain usable for several days.

The above mixture, in an .093 to .125 inch thickness did not cure completely at room temperature. Therefore thermal tests were also made placing a pressurized parabola in an oven maintained at 180°F. It was found that, if the temperature was raised slowly (within 1-2 hours) to 180°F, and then maintained at 180°F for two hours that a considerably strengthened parabola resulted. Inasmuch as such a condition would probably exist in space this procedure has been adopted. A rapid rise in temperature, on the other hand, resulted in a considerably deteriorated optical surface, due to warpage and "orange peel" formation.

Another disadvantage to the use of the Emerson and Cuming Eccospheres is the weight of the syntactic foam produced. These microballoons have a bulk density of 10-11 pound/cubic foot, approximately three times that of the phenolic microspheres.

In an attempt to make lower weight syntactic foams another type of glass spheres was obtained from the Hastings Plastics Company, of Santa Monica. These beads, trade named "Globe-O-Sil," had a density of approximately 3-1/2 pound/cubic foot. However, in tests with the Hetron 103 resin it was found that an adequate cure was not obtained, in thicknesses of approximately .100 inch,

when the samples were left in the sun for as long as 48 hours. This filler then either inhibited cure, or prevented adequate absorption of the UV radiation. Work was therefore discontinued on the polyester based syntactic foam.

#### POLYESTER RESINS

Since the use of Hetron 103 (a diallyl phthalate monomer resin) gave better optical results than the styrene modified materials it was decided to obtain a number of solid resins, and formulate materials using DAP as the reactive monomer. Three types of materials were used in these tests. These included a Wyandotte solid, polychloroester PR-2412, previously tested with styrene. Another type of resin was Food Machinery Corporation's DAPON "M," a partially polymerized diallyl phthalate. The third type of resins were isophthalic-maleic mixtures obtained from Diamond Alkali. In this case the isophthalic component is the low shrink member and the maleic resin is used to increase reactivity. The following resins were obtained from Diamond Alkali, (a) Dion-Iso #6421, a 1:1 mixture, (b) Dion-Iso #6424, a 1:2 mixture of isophthalic and maleic and Dion-Iso K-188, a 1:3 mixture.

The initial tests were made of the Wyandotte and Diamond Alkali resins using 40 percent DAP as the monomer with 2 percent benzoin and 2 percent benzoyl peroxide as the catalyst. The Wyandotte resins set up in approximately 24 hours out of doors, to a Barcol hardness of 35 to 40. The Dion resins after approximately 48 hours set up to a Barcol hardness of approximately 20 for the most reactive (K-188), to a hardness of 10 to 15 for the other resins. Since the Dion resins then apparently were not reactive enough no further work was done with these materials.

Additional tests were made with the FMC Dapon M resin and the Wyandotte resin using DAP monomer contents as low as 18 percent. In both cases the viscosity was extremely high so acetone was also used to reduce the viscosity so fabrics could be easily impregnated. Laminates made with either resin with a 25 percent content of DAP cured to

a Barcol hardness of approximately 30 to 40 in approximately 24 hours out of doors. Laminates were also made with 18 percent DAP content. With this low a monomer content the Dapon M materials only attained a hardness of approximately 10-20 in a two-day sunlight cure. The Wyandotte resins at this monomer content obtained a hardness of approximately 30.

Room temperature storage tests were also made of the Dapon M and the Wyandotte PR-2412 resin. Twenty-five gram samples of each catalyzed resin were stored with varying amounts of hydroquinone as an inhibitor. Inhibitor contents were approximately .015, .030, and .045 percent as well as noninhibited samples for controls. The Dapon M resins commenced gelation in one week, whereas the Wyandotte has so far been stable for approximately one month storage. Small sample laminates were made from time to time from the storage resins to check the activity. In these tests the Wyandotte materials appeared to set up satisfactorily in a 24-hour outdoor exposure.

# SHRINKAGE TESTS

An effort was made to determine the shrinkage exhibited by Fiberglass laminates made using Dapon M and the Wyandotte resin. These tests were made by laying up a 15 ply #103 glass fabric laminate on a Teflon coated, polished ferrotype plate. The plate had two sets of fine scratch marks placed, at 90 degrees to each other, seven inches and eight inches apart. By measuring the original distances on the plate very accurately, and then measuring the reproduced marks on the laminate, the coefficient of shrinkage could be calculated. Measurements were made of the laminate after room temperature UV curing and after a 16-hour elevated temperature cure. Table I shows the results of these tests.

	Seven-Inch	Dimension	Eight-Inch	Dimension
Material	Original	Post-Cure	Original	Post-Cure
	Shrinkage	Shrinkage	Shrinkage	Shrinkage
	Inches/	Inches/	Inches/	Inches/
	Inch	Inch	Inch	Inch
Wyandotte PR-2412 Dapon M Laminac 4123 (Control)	.0001	.00094	.0003	.0015
	.0016	.00190	.00216	.00235
	.0019	.00067	.00026	.00070

Table I. Polyester shrinkage tests.

Because of the lack of agreement between the two directions of measurement, in general and because of the conflicting measurements, i.e., the standard styrene based resin shrank less than either of the test resins, it was concluded that some error exists in the measurement. It should be pointed out that the laminate after fabrication is only approximately .033 inch thick and usually shows a small amount of warp. This then could contribute to the difficulty in making accurate measurements. Thicker laminates were not desired, however, since these would not be representative of the reinforcement actually used.

#### ADHESION TESTS

One of the major problems in the use of the polyester resins concerned the poor adhesion of the polyester resin to the Mylar surface. The use of a primer coat offered a considerable improvement in most cases. A series of tests were therefore made to determine which of three materials would be best for use. Flat panel specimens were made up using Wyandotte resin-glass cloth laminates laid upon a primed Mylar surface. Each laminate was  $3 \times 10$  inches with a 2 inch Mylar tab on each end. The Mylar was peeled from the laminate using a climbing drum peel test fixture in accordance with MIL-STD-401A, "Sandwich Constructions and Core Materials; General Test Methods." The primers tested and the results of the tests are shown in Table II.

Primer	Source	Average Peel Load, lb	Type of Failure
EC-776 46950	Minn. Mining & Mfg. Co.  E. I. du Pont de Nemours Co.	14.9 13.2	90% adhesive 100% cohesive
CS2414	Chem-Seal Corp.	21.5	100% cohesive

Table II. Polyester-primer adhesion tests.

From the results of the above tests it may be seen that the CS-2414 (a polysulfide) was considerably superior to the other materials. Unfortunately, in later tests the material was found very inconsistent, and did not show good strength at elevated temperatures. (The manufacturer claims this was due to change in formulation — Additional original formulation material will be supplied for test.)

#### POLYESTER PARABOLAS

In fabrication of parabolas two types of optical distortion are usually found. In those cases in which a fabric reinforcement is used the fabric pattern is usually visible, unless a relatively very thick gel coat is used. This effect has been found more pronounced when polyester resins are used than when epoxies are used. The effect is also more pronounced when a relatively thick, coarse weave fabric is used as compared to a thin, fine weave fabric. The reason for the fabric pattern visibility (which only becomes visible during and after cure) is the shrinkage of the resin into the fabric interstices. Thus a wet laminate, laid upon a glass plate, appears perfectly smooth at first. However, as the resin cures the shrinkage into the fabric interstices can be observed through the glass plate. Obviously the lower the resin shrinkage, the lower will be this effect. Other factors influencing this effect are pressure on the laminate, and the type and amount of reinforcement. Pressure applied during the cure cycle will force the laminate against the mold, and thus tend to "level out" the

hills and dales of the fabric. By the same token the use of relatively high amounts of fillers or reinforcements, which have lower shrinkage coefficients than the resin, will also tend to minimize shrinkage. Figure 1 illustrates a surface showing a fabric pattern.

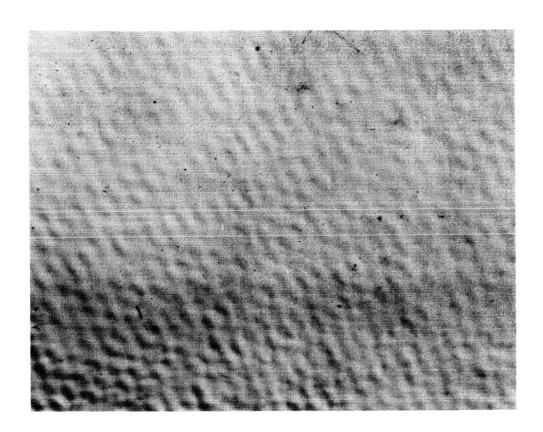


Figure 1. Mylar surface showing a fabric pattern.

The other type of optical distortion mentioned above is uneven waviness, and a so-called patternless distortion which also occurs, sometimes when the fabric pattern is present, but more often when it is not (see Figure 2). This patternless distortion is believed to be caused by uneven resin shrinkage before hardening takes place, and by unequal amounts of resin impregnation in the fabric reinforcement. Like the fabric pattern distortion this effect should also be minimized by pressure and relatively high amounts of filler, particularly if the



Figure 2. Patternless distortion surface, due to uneven shrinkage.

filler is high on a volume basis. The proof of this is that the parabolas made with microballoon fillers have been the best so far, due mainly to the high volume ratio of filler to resin.

In fabricating parabolas utilizing UV catalyzed polyester resins there are then a number of adverse factors which exert an influence against the possibility of obtaining a shrink-free laminate. First is the fact that in polymerization of polyesters the shrinkage is inherently high. Because of this, efforts are now being concentrated on utilization of resin systems which are known to have low shrinkage. One such system includes a high proportion of prepolymerized diallyl phthalate in the mixture. By using approximately 25 percent of liquid diallyl

phthalate the total shrinkage is cut down considerably. Another beneficial result of such a resin system, instead of the more conventional styrene monomer system, is a considerably lower rate of reactivity, which also reduces shrinkage. Another resin system being considered is a polychlorester, PR 2412, from Wyandotte, Since this is a very new product not much information is available, save that the maker claims it to have low shrinkage, a claim borne out by our preliminary tests.

Because of the mode of fabrication of the parabola it is impossible to exert pressure on the laminate, therefore no benefit is available from this process technique. The only other factors then which can be utilized are the incorporation of the finest weave fabric, and a high filler or reinforcement content which, however, must permit passage of UV radiation. This last requirement then severely limits the amounts and/or types of fillers usable.

With the above factors in mind a series of six-inch diameter parabolas were made using the 75 percent solid-25 percent liquid DAP monomer resin mixes, with #103 fiberglass as the reinforcement. In one series of tests samples were made using 5, 7, 10, 15 and 20 layers of #103 fabric and one layer of #120 fabric laid on a #2414 polysulfide primed surface. It was found that, using over 10 layers of fabric, practically no fabric pattern show through was visible. The patternless distortion, previously associated with polyester-fabric combinations, was also considerably reduced. The results were very similar with either the Wyandotte or the FMC Dapon-M resin. In all tests an effort was made to "rub out" the laminate to as low a resin content as possible. Resin content determinations were made of the 15 and 20 ply laminates and it was found they were 53 and 50 percent, respectively. Resin contents on previous samples had been found to run 55 to 60 percent.

Another significant test made was fabrication of three six-inch parabolas using in one case eight layers of a light Dacron marquisette fabric, another used six layers of #103 glass fabric and a third was divided into two halves; one side covered with a 15 percent Cab-O-Sil filled Wyandotte resin and the other with a 60 percent silica filled resin.

All of the samples cured satisfactorily, however the glass reinforced material appeared to cure the fastest. The optical surface of the glass reinforced material was also the best. The object of this test was to determine if the organic fabric, with its lower modulus, would show less distortion than the silica filled or glass reinforced fabric. Since the glass appeared best no further work was done with the other materials.

The results of the above tests then indicate that the use of thin fabric, low resin content and very even impregnation should result in the optimum laminate. Several one-foot diameter parabolas were made in the same manner. The optical results were somewhat mediocre, not as good as the six-inch parabolas. This was attributed to unevenness in impregnation. Figure 3 shows the best of these parabolas.



Figure 3. One-foot diameter Wyandotte polyester parabola with low resin content, fine weave cloth.

## EPOXY RESIN INVESTIGATION

#### SYNTACTIC FOAM

Previous tests had indicated that a syntactic foam, utilizing an epoxy resin and Union Carbide Company phenolic microballoons, gave an excellent optical surface. A number of one and two foot diameter parabolas were made using this material, in each case using either diethylenetriamine (DTA) or diethylaminopropylamine (Shell catalyst A) as the hardening agents. With DTA a working time of approximately 1 hour was the maximum whereas with hardener A a working time of approximately 3 to 4 hours was found. This working time, in these preliminary tests was very important since one of the major problems with the cpoxy syntactic foam was the development of a satisfactory technique to uniformly spread the foam over the Mylar surface. Previous techniques tried included hand spreading by trowels and spatulas, flattening with a roller, and pressing using a laminating press. This latter method was considered best, but not too practicable for five foot or larger diameter parabolas.

Two techniques have been developed which appear to be satisfactory. In one method a ball of foam paste is placed on top of the Mylar and a metal plate is placed on top of the foam and another under the Mylar. The entire assembly is then placed in a polyethylene bag and a vacuum is drawn. Depending on the stiffness of the foam the material can be forced down to a uniform thickness in one to three hours. Such a system can then be used for any size part, provided that a stiff enough plate is used, or that a large enough plate is available. The other system, which is also applicable to five foot or larger parabolas, consists of the use of a wedge shaped spreader bar. This bar functions similarly to a doctor blade, except that the bar, when being drawn across the Mylar, is held at a 45 degree angle to the surface. The effect then is to draw the paste in the direction of motion, but also as the bar moves the paste is forced down on the Mylar. Using this technique very uniform spreads have been obtained.

In addition to the spreading technique tests a series of samples was made to determine the optimum percent of microballoons to be used in the foam. It was found that approximately 50 percent by weight of phenolic microballoons could be incorporated into Epon 828 resin without the aid of a thinner such as acetone. However, such a mixture is extremely dry and is almost impossible to spread. When cured, this mixture has quite a low density and, because of its bulk has a relatively high stiffness-to-weight ratio. The optical surface, however, appeared slightly grainy. Mixtures made with as low as 15 percent microballoons, on the other hand are soft and are easily spread and have the disadvantage of flowing markedly before final cure. The optical surface with such a mixture appeared somewhat better than the highly filled mixtures. Samples were accordingly made up with 15, 22-1/2 and 30 percent of microballoon content. These mixtures were all applied to approximately 120 degree segments of a one-foot diameter parabola so as to obtain identical conditions in the Mylar, during cure, etc. After cure, the 22-1/2 percent mixture appeared to be the best from the standpoint of optics, and adhesion to the primed Mylar. This mixture then will be used for future tests.

In the development of techniques for spreading the syntactic foam it was found necessary to use a parting film between either the roller or a flat pressing plate in order to insure removal of either tool. However, because of the excessive tackiness of the foam it was also found extremely difficult to remove the parting film from the uncured foam. Films tried included polyethylene, polyvinylchloride, Mylar and cellophane. In attempting to pull off the film it was found that invariably large areas of foam would also be pulled off. A procedure was then developed where, using dry ice and acetone, the foam could be flash frozen, after which the film could be easily removed leaving a very smooth and uniform surface. The method then adopted is to first apply the syntactic foam uniformly to a circular area marked on a flat sheet of Mylar. The foam covered Mylar is then installed on the pressure fixture and first stretched and then held at the relaxed pressure, after which the foam is allowed to cure.

In all the syntactic foam tests made, as outlined above, room temperature hardening agents were used, mainly for convenience, until the fabrication details were developed. It was found that, using catalyst A, a parabola could be rigidized very satisfactorily by being maintained under pressure for 16 to 20 hours. However, such a procedure did not (in 20 hours) result in maximum strength. Post-cure treatments were then initiated, both to further strengthen the foam rigidized parabolas, and to determine the effect of heat on the structure. It was found that the parabolas could be heated as high as 240°F, without marked distortion, provided that the pressure was decreased during the cure so as to maintain a constant curvature. It was also found, that the temperature, on the materials tested, had to be gradually raised (at a rate of approximately 60 degrees/hour in order not to suffer excessive distortion.

In the initial heating tests severe delamination was found to take place. This was attributed to shrinkage in the Mylar. A number of tests were run on the Mylar film and it was found that shrinkages as high as 1.6 percent could be obtained in one hour at 325°F. At 250°F the shrinkage was approximately .68 percent in one hour with an increase to .82 percent in 16 hours. Since this last value represents almost an asymptotic value, this procedure will be used to "preshrink" all future Mylar used.

It would appear that the preshrinking operation should be done at 325°F, in order to secure maximum shrinkage. However, visual examination of the samples heat treated at 325°F indicated a slight diminishment of the brilliant luster, so the lower temperature will be used.

Epoxy syntactic foam parabolas using preshrunk Mylar then showed much less evidence of severe delamination after a post-cure heat treatment. However, in most cases a post-cure of such parabolas resulted in production of a great many blisters appearing on the surface of the Mylar. It was surmised that these blisters could have resulted from possibly three causes; (a) air bubbles in the foam which expanded under heat, (b) poor surface preparation of the Mylar so the primer did

not hold or (c) a poor bond between the primer and the reinforcement. In some cases it was believed the blisters may have been the result of a combination of all three causes plus additional stresses set up the Mylar by the heat treatment. Figure 4 illustrates a typical blistered section.

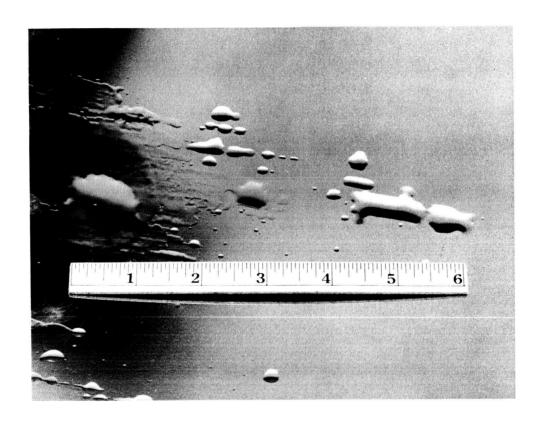


Figure 4. Typical blistered parabola.

Examination of some of the blistered parabolas showed that in a few cases air bubbles had been entrapped between the foam and the Mylar film. Subjecting the foam to a vacuum prior to installation effectively removed most of the air. Parabolas made with evacuated foam were improved somewhat with regard to blister formation, but all the post-cure blisters were not removed by this treatment.

#### ADHESION TESTS

A number of special tests were made on flat Mylar panels in an attempt to isolate the causes for the major portion of the blisters. One set of tests was made to determine the effect on blistering and the differences in adhesion of the foam to three types of surfaces. This test was also designed to test the effect of several process conditions. The test was made as follows: a 4 ft. x 16 inch sheet of preshrunk Mylar was cleaned with reagent acetone, primed with DuPont #46950 primer plus 3 percent of curing agent 805. After air drying the primer overnight it was oven cured at 300°F for three minutes. The sheet was then divided into two sheets, A and B. One-half of sheet A was coated with a thin coat of DTA catalyzed Epon 828 and allowed to cure overnight. Sheet B, the next day, was coated over one-half its surface with a thin coat of a similar epoxy resin. Both sheets were then immediately coated with a layer of syntactic foam made to the following formula:

Epon 828

- 100 parts

Phenolic microballoons - 22-1/2 parts

- LL-1/L part.

Cab-O-Sil

4 parts

Catalyst A

8 parts

This test then gave two sheets of material, essentially identical, except for three different prime coats; #46950 plus 805 curing agent, #46950 plus a cured epoxy coat and #46950 plus a wet epoxy coat. After foam coating, each sheet was cut into four parts. Each part was then subjected to a different processing treatment ranging from a simple room temperature cure to a vacuum cure plus a post-cure in vacuum. Table III shows the various types of processing treatments used to cure the foam, and the results secured from the standpoint of blisters and adhesion. The presence or absence of blisters was observed visually. Adhesion, however, was checked by attempting to peel the two mil Mylar away from the hardened foam reinforcement.

As a result of the foregoing it was concluded that the use of an epoxy prime coat was probably more beneficial than the use of the #46950 primer, particularly when used with the epoxy foam. Furthermore, it was very evident that room temperature cure followed by a

			Results	lts	
		Blisters	ters	Adhesion	sion
Sample No.	Processing Treatment	#46950 Surface	Epoxy Surface	#46950 Surface	Epoxy Surface
1A 1B	48-hour room temperature cure 48-hour room temperature cure	None None	None None	Poor	Fair Poor
2A 2B	Room temperature cure plus 3 hr. 180°F post-cure Room temperature cure plus 3 hr. 180"F post-cure	Blisters Blisters	None None	Fair Fair	Good
3A	4.	Blisters	Blisters	Fair	Fair
3B	pressure cure plus 3 hr. 180° F post-cure Evacuated form, 48 hr. room temperature and pressure cure plus 3 hr. 180° F post-cure	Blisters	Blisters	Fair	Good
4A 4B	Evacuated foam, 3 hr. cure at 180°F in vacuum Evacuated foam, 3 hr. cure at 180°F in vacuum	None None	None None	Fair Fair	Good
			1 1 1 1		

NOTES: 1) All "A" samples had a cured epoxy prime coat. All "B" samples had a wet epoxy prime coat.

Good adhesion meant that 70 to 100 percent of the Mylar adhered to the cured foam.  $^{\circ}$ Poor" adhesion meant that the Mylar could be readily pulled away. "Fair" adhesion meant approximately 30 to 60 percent of the Mylar adhered to the cured foam. 2)

Blister and adhesion tests. Table III. post-cure was not a beneficial procedure. Thirdly, it appeared that heat curing of the foam under vacuum conditions resulted in the best part. Since this latter procedure was, in fact, what would be used in production of the actual parabola, in space, it was concluded that the majority of future tests should be run in this manner.

In order to verify the results and conclusions reached above, and to test various cleaning techniques a few more tests were run, using the "old" room temperature cure and post-cure technique. In these tests an 18 x 18 inch piece of 2 mil Mylar was divided into three parts. One part was well scrubbed with Ajax household cleaner and water followed by a distilled water rinse. A second section was cleaned with Bloomingdale Rubber Company Pre-bond 700, a special cleanser used in preparation of stainless steel prior to bonding, and the third section was scrubbed with several applications of reagent acetone. Half of each panel was coated with #46950 plus curing agent 805 and half was coated with Epon 828 plus 10 percent catalyst A, laid directly on the Mylar, and allowed to cure overnight. All panels were covered with approximately 1/8 inch of syntactic foam using 30 percent of phenolic microballoons and 8 percent DTA as the catalyst. The panels were room temperature cured for 16 hours and were then given a three-hour postcure at 180°F.

Another set of panels was made exactly as above except that the foam was made using catalyst A instead of DTA. The results of both series of tests are shown in Table IV.

The results shown in Table IV, while contradictory in the adhesion results, do indicate that both the Ajax cleanser or the Pre-bond 700 are better than the use of acetone. It also indicated that, with catalyst A, an excellent bond could be achieved from the foam to the Mylar, without the need for a special primer coat. It is surmised that the difference in adhesion shown by the two foams is due to better wetting of the Mylar by the slower curing catalyst A.

				Results	ults	
			Blisters	SIS	Adh	Adhesion
Sample No.	Cleaning Technique	Curing Process	#46950 Surface	Epoxy Surface	#46950 Surface	Epoxy Surface
1 (DTA) Ajax	Ajax	Room temp. plus 180°F post-cure	Moderate	Slight	Good	Fair
2 (DTA)	2 (DTA) Pre-bond 700	Room temp. plus 180°F post-cure	Moderate	Slight	Good	Fair
3 (DTA)	3 (DTA) Acetone	Room temp. plus 180°F post-cure	Moderate	Slight	Fair	Poor
4 (A)	Ajax	Room temp. plus 180°F post-cure	Slight	None	Poor	Very good
5 (A)	Pre-bond 700	Room temp. plus 180°F post-cure	Slight	None	Poor	Very good
(A)	Acetone	Room temp. plus 180°F post-cure	Slight	None	Poor	Good

Table IV. Cleaning techniques tests.

#### "SPECIAL" EPOXY RESIN TESTS

All of the above tests were made using resin systems utilizing room temperature catalysts. Such catalysts would, of course, not be practical for ultimate usage because of their extremely limited shelf life. An investigation was therefore made into epoxy resin systems which could be catalyzed and which would remain flexible for at least one month at room temperature, and which could then be cured in a moderate time at temperatures not exceeding 240°F. Currently, two such systems are available. One system has recently been produced by the General Mills Corporation. Two samples of this type of material have been procured, XW-100 and XW-101. Both materials are one component materials with a claimed shelf life of at least two months at room temperature. In the first preliminary tests good laminates were made by heating for approximately 24 hours at 250° and for approximately 72 hours at 180°F. While these cure times may be somewhat excessive, the material does appear to offer promise of being usable for this application. Since both materials were supplied with approximately 50 percent of thinner incorporated into the resin, neither was judged too suitable for space application. Consequently, no syntactic foams have been made with either resin. A request has been made to the vendor for unthinned resin.

The second system being investigated concerns the use of a new catalyst being produced by the U.S. Borax Company. This catalyst, USB 110, a borate,  $2-(\beta-\text{dimethylaminoethoxy})-4$  methyl 1, 3, 2-dioxaborinane, can be mixed with the resin with no reaction at room temperature, other than a slight viscosity change. Preliminary tests with the catalyst and two resins, Shell Chemical Company Epon 828, and Union Carbide Company ERL 2795, indicate that at 240°F cure takes place in approximately 24 hours and approximately 48 hours at 200°F. Further, quantitative tests are also planned for this system.

#### MYLAR FILM TESTS

As a consequence of the various operations which are performed on the Mylar it was found virtually impossible to avoid damage to the aluminized surface. A protective strippable coating, Spraylat SC-1071 from the Spraylat Corporation, was found to give excellent protection to the surface. This material, however, will not withstand the preshrink heat treatment operation. It will therefore be requested to be applied to the film immediately after pre-shrinking by the G.T. Schjeldahl Co., and will be left on during the mosaic fabrication and the parabola fabrication. It is believed this should result in a considerable improvement of the final product.

Another test made with the Mylar consisted of making several parabolas with three mil material, instead of two mil. The use of the thicker Mylar appears to result in a better optical surface. More tests are planned with this material and also five mil material, which at present is not available.

#### FUTURE PLANS

For the next quarter it is planned to investigate the following:

- 1. Additional elevated temperature tests will be made on small parabolas in a vacuum, to develop the optimum fabrication technique for each system. In particular it is planned to investigate the use of a silicone RTV adhesive as a heat resistant primer for the polyester. In conjunction with the primer it is planned to examine nylon stretch fabric, felts and similar loose fabrics as mechanical lock fabrics to achieve improved adhesion at elevated temperatures.
- 2. Additional tests will be made to develop an optimum epoxy resin which cures at 200 240 °F and still has good catalyzed shelf life.
- 3. Erection and rigidization tests will be initiated on the full size parabola.
- 4. Flat panel specimens will be prepared for mechanical, thermal and radiation tests.